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Mechanical Properties of Degraded PMR–15 Resin

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MECHANICAL PROPERTIES OF DEGRADED PMR-15 RESIN

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SUMMARY

Thermo-oxidative aging produces a nonuniform degradation state in PMR-15 resin. A surface layer, usually attributed to oxidative degradation, forms. This surface layer has different properties from the inner material. A set of material tests was designed to separate the properties of the oxidized surface layer from the properties of interior material. Test specimens were aged at 316 °C in either air or nitrogen, for durations of up to 800 hr. The thickness of the oxidized surface layer in air aged specimens, and the shrinkage and coefficient of thermal expansion (CTE) of nitrogen aged specimens were measured directly. Four-point-bend tests were performed to determine modulus of both the oxidized surface layer and the interior material. Bimaterial strip specimens consisting of oxidized surface material and unoxidized interior material were constructed and used to determine surface layer shrinkage and CTE. Results confirm that the surface layer and core materials have substantially different properties.

INTRODUCTION

Background

Polymer matrix composite materials are being increasingly considered for use in environments that challenge their durability. Such applications include turbine engine structures and high speed aircraft skins. In these environments the materials are exposed to high temperature and to oxygen, both of which contribute to the degradation of the polymer matrix.

Significant progress has been made in the understanding of the aging effects on the thermo-oxidative stability of polymer matrix composites (refs. 1 to 3). Much of this work has focused on PMR-15 resin as a representative material. Thermo-oxidative aging produces a nonuniform degradation state in PMR-15 resin. While thermal degradation occurs throughout the material, oxidative degradation occurs only where oxygen diffuses into the material. This produces an oxidized surface layer that has different properties from the unoxidized inner material. Current models of coupled diffusion-reaction mechanisms (refs. 4 to 6) attempt to capture the behavior of this chemical degradation. However, there is a need for data on material properties of degraded material(s), in order to link the diffusion-reaction models of chemical degradation to thermo-mechanical models (ref. 7).

Air aged material specimens have an oxidized surface layer and an unoxidized inner material which have different material properties. Tests performed on such specimens as if they were homogeneous produce results that are difficult to interpret and may have little meaning. Tests must be carefully designed to separate the properties of the surface layer from the properties of the inner material.

Problem

The problem addressed here is to separate the properties of the oxidized surface layer from the properties of the unoxidized inner material, in PMR-15 resin specimens aged in air for various times at 316 °C. Specifically, the thickness of the oxidized surface layer and the modulus, CTE, and shrinkage of both surface and inner material are determined as functions of aging duration.

Approach

The approach to this problem is primarily experimental. The main challenge of this project, and the problem that drove the experimental design, is how to determine the shrinkage and CTE of the oxidized surface layer. The shrinkage is small, and the changes are slight. Typical measurements of length are confounded with the effects of surface erosion on the ends, and the stress and strain interactions of the surface layer and the unoxidized inner material. In order to determine the surface layer shrinkage and the surface layer CTE, a new test based on a bimaterial strip model was designed and used.

The new test involved the manufacturing of specimens that resembled a bimaterial strip, with the oxidized surface material on one side, and unoxidized inner material on the other side. These specimens were manufactured simply by slicing a piece of an aged specimen lengthwise through the thickness. When one side contracts or expands relative to the other, the specimen curves. By utilizing a bimaterial strip model, the amount of curvature and the change in curvature with temperature can be used to determine the surface layer shrinkage and the CTE. An illustration of the curvature specimen is shown in figure 1.

In order to calculate the surface layer shrinkage and CTE from the curvature and change in curvature with temperature, several other properties must be known. These include surface layer thickness, modulus of both the oxidized surface layer and unoxidized material, shrinkage of unoxidized aged material, and CTE of unoxidized aged material. These properties are determined from other tests.

Surface layer thickness is measured simply by polishing a cross section of a specimen and measuring the surface layer. The modulus of unoxidized aged material is determined by standard four-point bend test, which is illustrated in figure 2. The modulus of the oxidized surface layer is derived using basic beam theory from a bend test of an air aged specimen, as surface layer thickness and the modulus of the unoxidized material are known. Figure 2 also illustrates the bend test of an air-aged specimen. Shrinkage of unoxidized aged material is measured simply by measuring the length of a specimen both before and after aging. The CTE of unoxidized aged material is determined from a typical test using thermo-mechanical analysis, illustrated in figure 3.

EXPERIMENTAL PROCEDURE

Overview

Specimens were cut from plaques of PMR-15 resin. Each specimen was then measured to determine initial dimensions. After initial measurements were completed, the specimens were aged in an oven at 316 °C. Half of the specimens were aged in air in the oven, and half were aged in nitrogen in a nitrogen chamber placed inside the oven. Once specimens were removed from the aging ovens, their dimensions were remeasured. They were then tested to determine modulus by using four point bend tests. After bend testing, the specimens were cut again into smaller specimens for bimaterial strip curve tests, thermomechanical analysis (TMA), and for measuring surface layer thickness. Typically five specimens were used per test.

Specimen Preparation

These experiments used 152.4 by 152.4 mm PMR-15 neat resin plaques from the same batch as the resin plaques used by Kamvouris and Roberts (ref. 8), so that comparisons could be made. The plaques were made from HyComp 100, a PMR-15 powder supplied by HyComp, Inc. The powder was compression molded using an automated heated press with vacuum. Both temperature and pressure were ramped gradually, to a 2-hr hold at 316 °C and 9.3 MPa. The material was not given a separate post cure before aging.

Specimens were cut from the plaques using a water-cooled micromachining diamond saw. The specimens were cut to the dimensions specified for four-point bend testing in the ASTM Standard Test Method for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials (D 790M). Specimens measured approximately 54- by 10- by 2.5-mm. After aging, dimensional measurements, and bend testing were completed, the specimens were further cut into smaller pieces for use in other tests. These pieces are illustrated in figure 4. Long thin specimens approximately $53 \times 3 \times 1$ mm were cut for use in bimaterial strip curvature tests. Specimens approximately $26 \times 3 \times 2.5$ mm were cut for determining layer thickness. Specimens approximately $5 \times 3 \times 2.5$ mm were cut for use in thermomechanical analysis.

Specimens were dried for 24 hr at 120 °C and stored in sealed plastic bags inside a dessicator, and were dried again before each measurement for at least 30 min in a 120 °C oven, except for the TMA specimens, which were dried for two or more hours.

Specimens were aged in a Blue M oven at 316 °C. Specimens aged in air were placed on a tray in the oven, while specimens aged in nitrogen were placed inside a nitrogen chamber inside the oven. Specimen groups were aged 24, 48, 96, 168, 240, 336, 465, 633, and 801 hr. One group was not aged.

Tests

Nitrogen Aged Specimen Shrinkage.—Shrinkage of nitrogen aged specimens was determined from direct measurements of the length of the bend test specimens before and after aging. Measurements were made using a traveling measuring microscope, which measured to 0.001 mm. The coordinates of each corner were recorded for both the top and bottom of each specimen. This produced four measurements of length for each specimen. The shrinkage could then be determined by comparing the length of each specimen before aging to the length after.

Bend Tests.—An Instron 4505 load frame with a 4500 controller was used for the bend tests. Steel four point bend fixtures with ceramic rollers were used. The load span to support span ratio was two. The bend tests conformed to ASTM D 790M, with one exception. Because of difficulties obtaining an accurate means of measuring center point deflection, load frame crosshead deflection was used instead. To compensate for slack and compliance of the load train, a test with a rigid bar in the fixture was performed to determine the compliance of the system. This was then used to correct the load versus displacement data for the tested specimens. Specimens were not tested to failure.

Thermomechanical Analysis.—Thermomechanical analysis was used to determine the CTE of nitrogen aged specimens. A TA Instruments model 2940 was used to measure the expansion of the small specimens as temperature was increased. CTE was determined from the slope of expansion versus temperature, over the range of temperature from room temperature to 316 °C.

Surface Layer Thickness.—Surface layer thickness specimens were mounted in epoxy and the cross section of the specimen was polished, to enable viewing of the surface layer with microscopy. Photomicrographs were then taken of the specimens. The thickness of the surface layer was then determined from measurements taken from the photomicrographs. This method of examination was known to produce measurable results due to the experience of Bowles, using similar material and aging conditions (ref. 9).

Curvature Tests.—Specimen curvature at room temperature was determined by measuring the location of three points on the edge of the curvature specimens, using a travelling measuring microscope that measured to 0.001 mm. Measurements were taken on both sides of each specimen. To make measurements at elevated temperatures, specimens were placed in the oven of the Instron 4505 test machine. Measurements were made using a cathetometer that looked through the oven window at the specimens. Specimens were placed on small stands in the oven, and measurements were taken of the height and span of the arc, as shown in figure 1.

ANALYSIS

Assumptions

The following analysis rests on a few basic assumptions. First is that the surface layer has uniform properties throughout the layer. This assumption is supported by the work of Cunningham, who showed both experimentally and with models that the surface degradation occurs on a sharp front, with little apparent gradient in degradation level within the surface layer (ref. 10). Second is the assumption that the moduli of the surface and interior layers have the same temperature dependence. This assumption is as yet unsupported, but effects only the surface layer CTE calculations in this work. Third is the assumption that viscoelastic relaxation is negligible. Relaxation would tend to make measurements of shrinkage underestimates of actual shrinkage. Finally, it is assumed that the nitrogen-aged specimens and the apparently unoxidized core material in the air aged specimens have the same properties.

Nitrogen Aged Specimen Shrinkage

The shrinkage of a nitrogen aged specimen, which represents the shrinkage of unoxidized aged resin, is simply as follows:

$$\varepsilon_{sc} = \frac{\left(l_{aged} - l_0\right)}{l_0} - \left(\alpha_{c(aged)} - \alpha_{c(o)}\right)\Delta T \qquad (1)$$

where ε_{sc} is the shrinkage strain of the unoxidized material, l_{aged} is the aged length, and l_0 is the unaged length. The second term compensates for the small difference between the aged and unaged core material CTE's, $\alpha_{c(aged)}$ and $\alpha_{c(o)}$, with ΔT being the difference between the aging temperature and room temperature.

Bend Tests

The modulus of nitrogen aged specimens, E_c, was calcualted according to ASTM D 790M:

$$E_c = \frac{0.17L^3 m}{wt^3} \qquad (2)$$

where L is the support span, w is the specimen width, t is the specimen thickness, and m is the slope of the tangent to the initial straight-line portion of the load-deflection curve. For the specimens tested, the load-deflection curves were very linear.

Modulus of the oxidized surface layer, E_s, was determined by using basic beam theory:

$$E_s = \frac{1}{I_s}\left(\frac{L^3 m}{96} - E_c I_c\right) \qquad (3)$$

where L is the support span, m is the slope of the tangent to the initial straight line portion of the load deflection curve, and:

$$I_c = \frac{\left(w - 2t_s\right)\left(t - 2t_s\right)^3}{12} \qquad (4)$$

$$I_s = \frac{wt^3}{12} - I_c \qquad (5)$$

where t is the total thickness, and t_s is the thickness of the surface layer.

Thermomechanical Analysis of Nitrogen Specimens

CTE of the unoxidized core material was determined from thermomechanical analysis of nitrogen aged specimens. The TMA testing machine determined the slope of dimensional change versus temperature. From there calculating the CTE was simply:

$$\alpha_c = \frac{m_{CTE}}{t} \qquad (6)$$

where α_c is the coefficient of thermal expansion and m_{CTE} is the slope of the dimension change versus temperature.

Surface Layer Thickness

Surface layer thickness was determined by optical microscope measurement of prepared cross sections. Calculations were only to convert scale.

Curvature Tests

The curvature tests are based on a model of a bimaterial beam, as illustrated in figure 1. Since the beam is unrestrained, the moment balance is of the form:

$$M = 0 = \int \sigma z \, dz \qquad (7)$$

where the stress includes a thermal expansion term and a shrinkage term:

$$\sigma = E(\varepsilon - \alpha \Delta T - \varepsilon_s) \qquad (8)$$

Using Bernoulli-Euler beam theory, the strain term can be expressed as:

$$\varepsilon = kz + \varepsilon_0 \qquad (9)$$

where k is the curvature and ε_0 is the strain at the neutral axis. Thus stress can be expressed as

$$\sigma = E(kz + \varepsilon_0 - \alpha \Delta T - \varepsilon_s) \qquad (10)$$

and the moment is then of the form:

$$M = \int E(kz + \varepsilon_0 - \alpha \Delta T - \varepsilon_s) z \, dz \qquad (11)$$

It would be desirable to transfer coordinates to the neutral axis before integrating. The location of the neutral axis, as shown in figure 5 is

$$z^* = \frac{E_s \left(t_c + \dfrac{t_s}{2} \right) t_s + E_c \left(\dfrac{t_c}{2} \right) t_c}{E_s t_s + E_c t_c} \qquad (12)$$

where t_s is the thickness of the surface layer and t_c is the thickness of the unoxidized core material. Establishing the new coordinate system we have:

$$\begin{aligned}
\zeta_1 &= t_s + t_c - z^* \\
\zeta_2 &= t_c - z^* \\
\zeta_3 &= -z^*
\end{aligned} \qquad (13)$$

Thus the moment balance equation becomes:

$$M = 0 = \int_{\zeta_2}^{\zeta_1} E_s (k\zeta + \varepsilon_0 - \alpha_s \Delta T - \varepsilon_{ss}) \zeta \, d\zeta + \int_{\zeta_3}^{\zeta_2} E_c (k\zeta + \varepsilon_0 - \alpha_c \Delta T - \varepsilon_{sc}) \zeta \, d\zeta \qquad (14)$$

Where α_s and α_c are the surface layer and core CTE's respectively, and ε_{ss} and ε_{sc} are the surface layer and core shrinkage strains. Curvature k can be determined from measurements as:

$$k = \frac{8h}{s^2} \qquad (15)$$

Where h is the height of the arc and s is the span. Plotting values of k versus temperature and fitting a line to the data produces a graph like figure 6, where k' is the slope of k versus T and k" is the curvature at the cure temperature of 316 °C, where ΔT is zero. Thus k can be expressed as:

$$k = k' \Delta T + k" \qquad (16)$$

Integrating and simplifying the moment balance equation leads to:

$$M = 0 = \frac{1}{3}aE_sk + \frac{1}{3}bE_ck - \frac{1}{2}cE_s\varepsilon_{ss} - \frac{1}{2}dE_c\varepsilon_{sc} - \left[\frac{1}{2}cE_s\alpha_s + \frac{1}{2}dE_c\alpha_c\right]\Delta T \qquad (17)$$

where

$$
\begin{aligned}
a &= \zeta_1^3 - \zeta_2^3 \\
b &= \zeta_2^3 - \zeta_3^3 \\
c &= \zeta_1^2 - \zeta_2^2 \\
d &= \zeta_2^2 - \zeta_3^2
\end{aligned}
\qquad (18)
$$

and, as a consequence of the choice of axis,

$$cE_s = -dE_c \qquad (19)$$

For $\Delta T = 0$, we can find

$$M = 0 = \frac{1}{3}aE_sk'' + \frac{1}{3}bE_ck'' - \frac{1}{2}cE_s\varepsilon_{ss} - \frac{1}{2}dE_c\varepsilon_{sc} \qquad (20)$$

Solving for surface layer shrinkage:

$$\varepsilon_{ss} = \frac{\frac{2}{3}aE_sk'' + \frac{2}{3}bE_ck''}{cE_s} + \varepsilon_{sc} \qquad (21)$$

Now for arbitrary ΔT it is possible to determine the coefficient of thermal expansion of the surface layer material. Inserting equation (19) into equation (17) and solving:

$$\alpha_s = \frac{\frac{2}{3}aE_sk + \frac{2}{3}bE_ck}{cE_s\Delta T} - \frac{\varepsilon_{ss}}{\Delta T} + \frac{\varepsilon_{sc}}{\Delta T} + \alpha_c \qquad (22)$$

It is convenient to present shrinkage strain ε_s as a percent shrinkage P_s; this can be calculated simply from:

$$P_s = -100\varepsilon_{ss} \qquad (23)$$

RESULTS AND DISCUSSION

Format for Plotting

Data for each specimen was reduced individually. Results from replicate specimens (typically five per condition but occasionally as few as three) were then averaged, and the standard deviation were calculated. Symbols in the following plots indicate the mean, and error bars represent plus-minus one standard deviation.

Surface Layer Thickness

Surface layer thickness are shown in figure 7. Surface layer thickness increases with aging duration, but the rate of growth decreases. The curve very closely resembles similar measurements made by Bowles (ref. 9).

Modulus

The results of the bend tests are shown in figure 8. The measured value of the stiffness for the nitrogen aged specimens remained rather constant regardless of aging duration. The calculated value for the stiffness of the oxidized surface layer indicates a significant increase in the modulus over that of unoxidized material. However, the modulus of oxidized material also remains rather constant with aging duration.

Surface Layer Shrinkage

Shrinkage of nitrogen aged specimens and calculations of the shrinkage of the oxidized surface layer are shown in figure 9. Values for strain were derived using equations (1) and (21), then converted to percent shrinkage for plotting using equation (23). The plots indicate that oxidized surface layer shrinkage is significantly greater than that of the inner unoxidized material. Shrinkage increases as aging duration increases although the unoxidized nitrogen aged specimen shrinkage appears to approach a limit as aging duration increases. Oxidized surface layer shrinkage does not appear to have a similar limiting behavior.

Coefficient of Thermal Expansion

Thermomechanical testing was used to determine CTE of nitrogen aged specimens. The CTE of the oxidized surface layer was derived from curve test results as was shown in equation (22). A plot of CTE versus aging duration is shown in figure 10 for nitrogen aged specimens and for the calculated value of the oxidized surface layer. The data indicates that the CTE of material aged in nitrogen decreases slightly as time increases. The calculated CTE of the oxidized surface layer decreases more markedly.

DISCUSSION AND CONCLUSION

This set of experiments successfully provides separate properties for the surface layer and the inner material of aged PMR-15 resin. These properties will be valuable for both greater understanding of aged material behavior, and also as data that can be used to complete models of thermo-oxidative degradation.

The core and surface layer material prove to be notably different. This difference explains some of the complex observed behavior of thermo-oxidatively aged specimens. For example, it was observed that curvature specimens at room temperature curved towards the interior material, when it was initially assumed that the specimens would curve toward the shrunken surface layer. Curvature specimens at the aging temperature curve toward the surface layer. Changes in CTE seen in this experiment explain this behavior. The increased stiffness and shrinkage seen in the surface layer also helps to explain surface cracking observed in other investigations in air-aged specimens. The shrinkage of the surface is restrained by the core, resulting in tensile stresses, which are only enhanced by the material's increased modulus.

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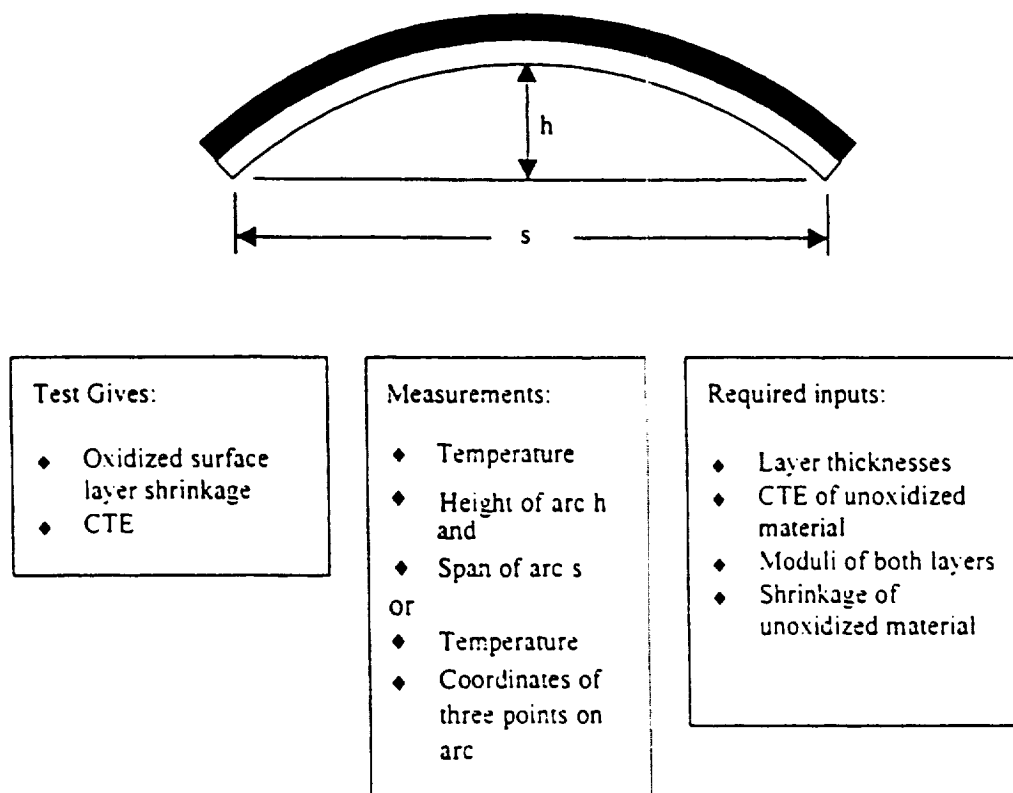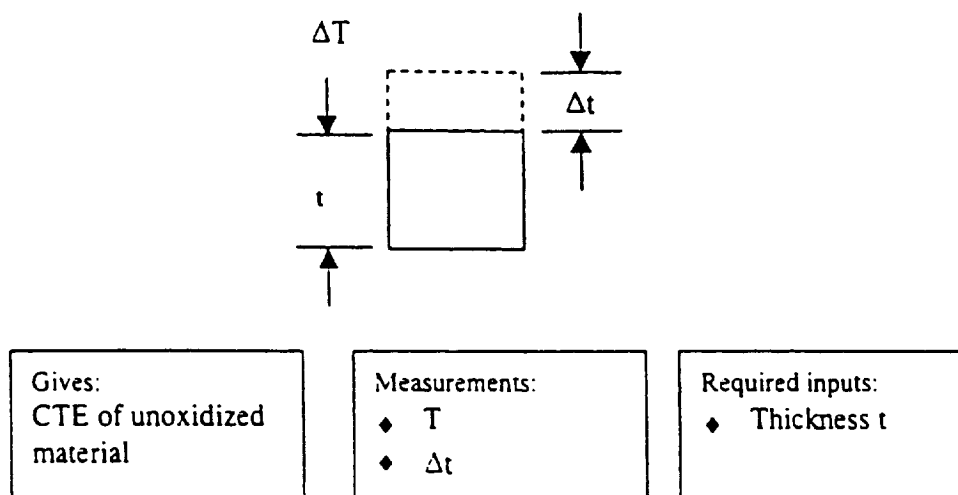
Figure 1.—Curve test schematic.

Unoxidized material

| Gives:
Modulus of
unoxidized material | Measurements:
♦ Load
♦ Displacement | Required inputs:
♦ Thickness |

Oxidized material

| Gives:
Modulus of oxidized
material | Measurements:
♦ Load
♦ Displacement | Required inputs:
♦ Thickness of
surface layer
♦ Thickness of
specimen
♦ Modulus of
unoxidized
material |
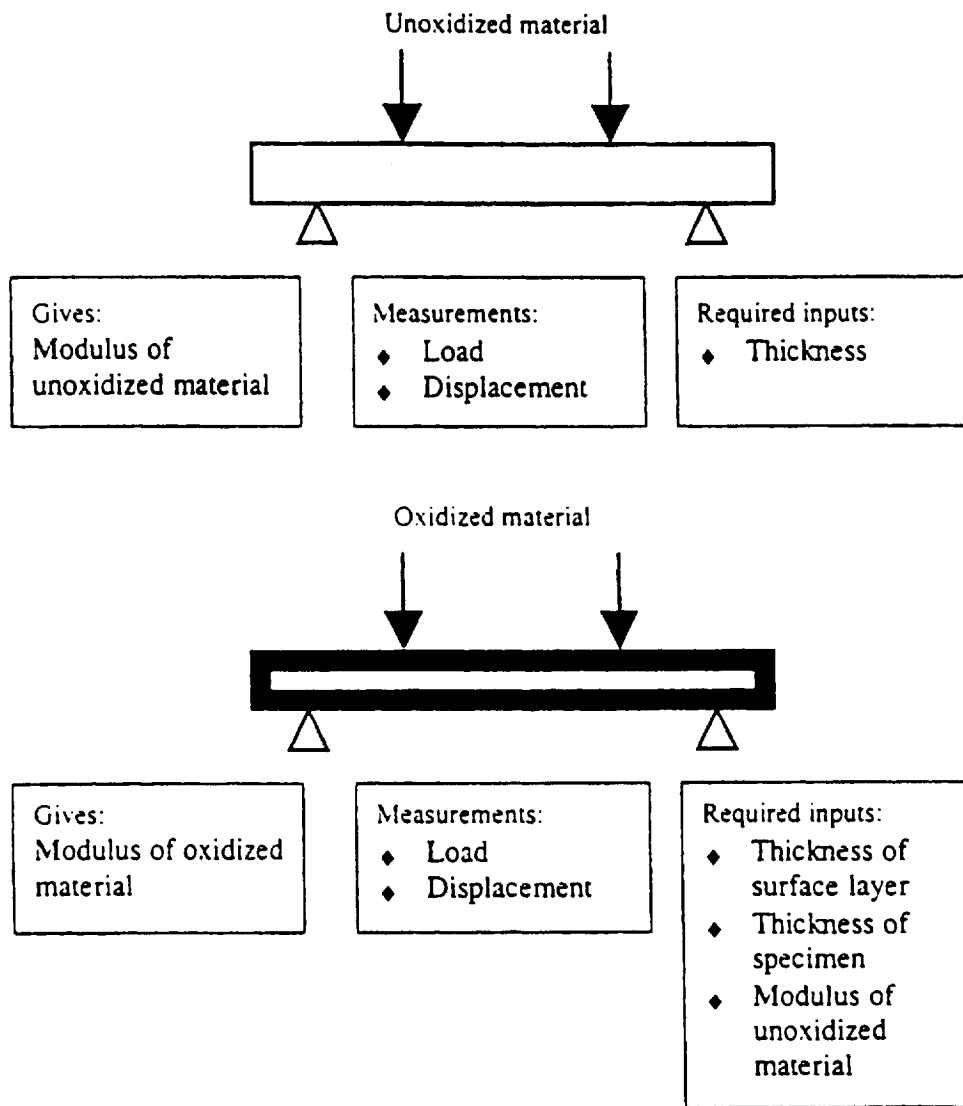
Figure 2.—Four-point bend test schematics.

ΔT Δt

t

| Gives:
CTE of unoxidized
material | Measurements:
♦ T
♦ Δt | Required inputs:
♦ Thickness t |

Figure 3.—CTE test schematic.

A	Original 4-point bend test specimen
B	Spare Material
C	Curve Test Specimen
D	Extra Curve Test Specimen
E	Surface layer thickness specimen
F-H	Thermo-mechanical test specimens
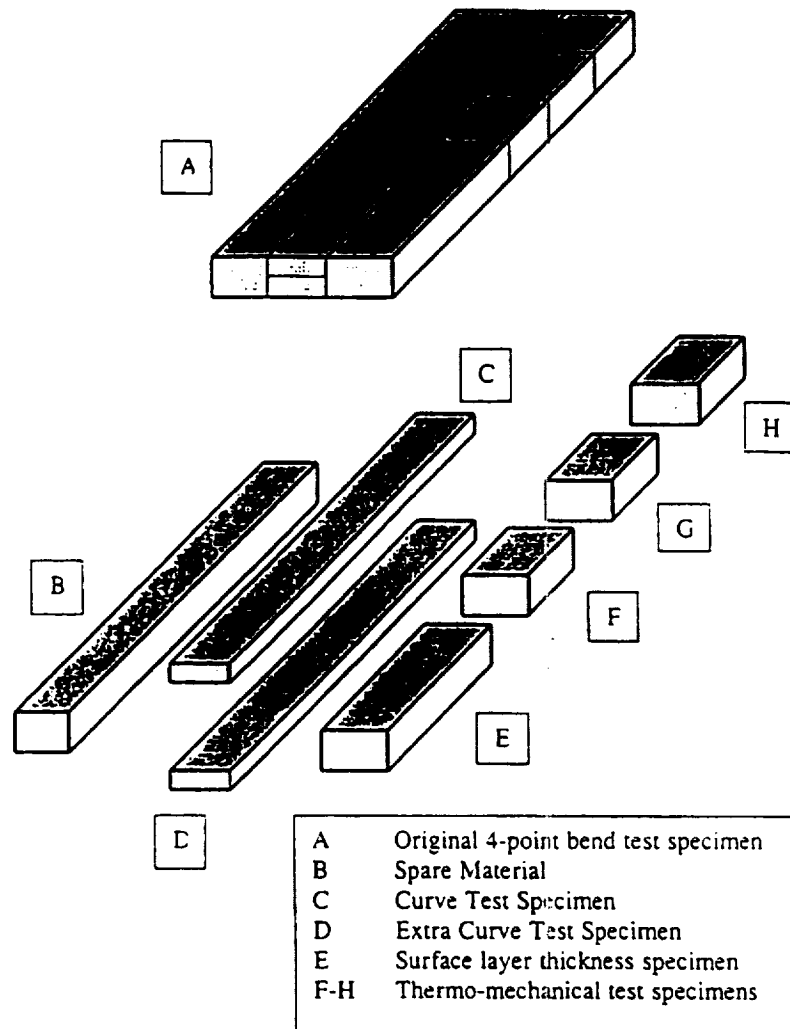
Figure 4.—Specimen cutting illustration.
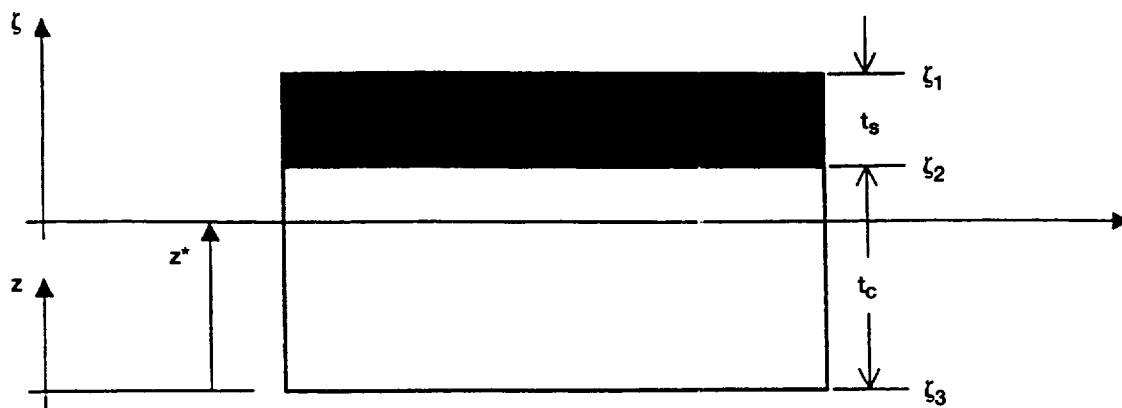
Figure 5.—Neutral axis and coordinate system.
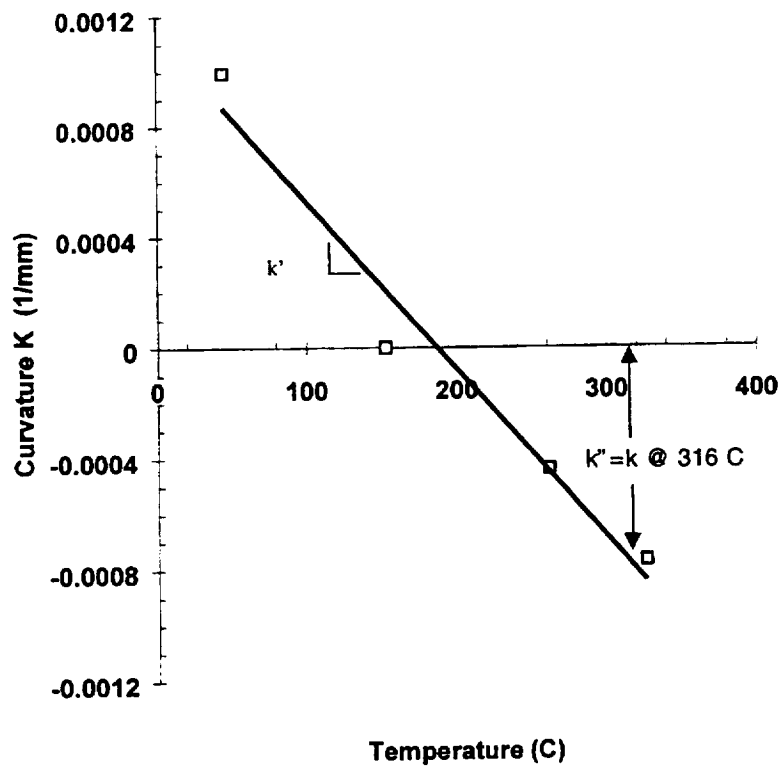
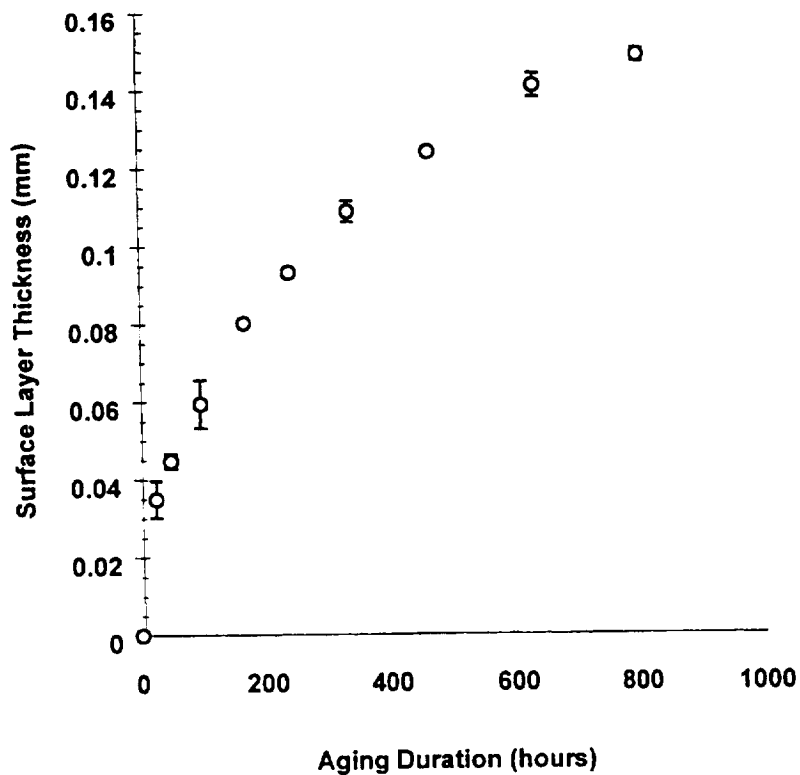Figure 6.—Sample graph of curvature versus temperature, showing k' and k".

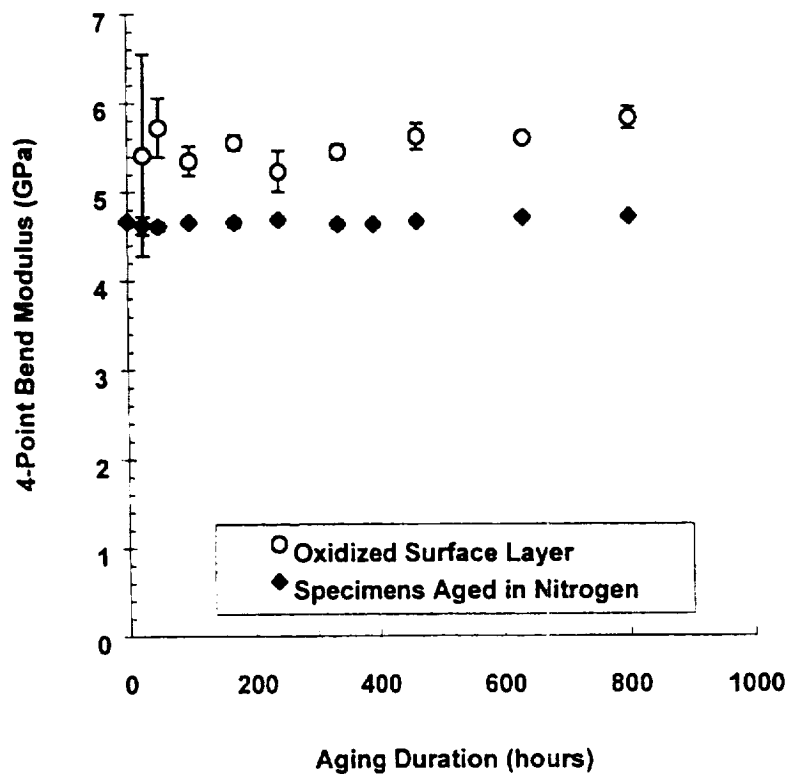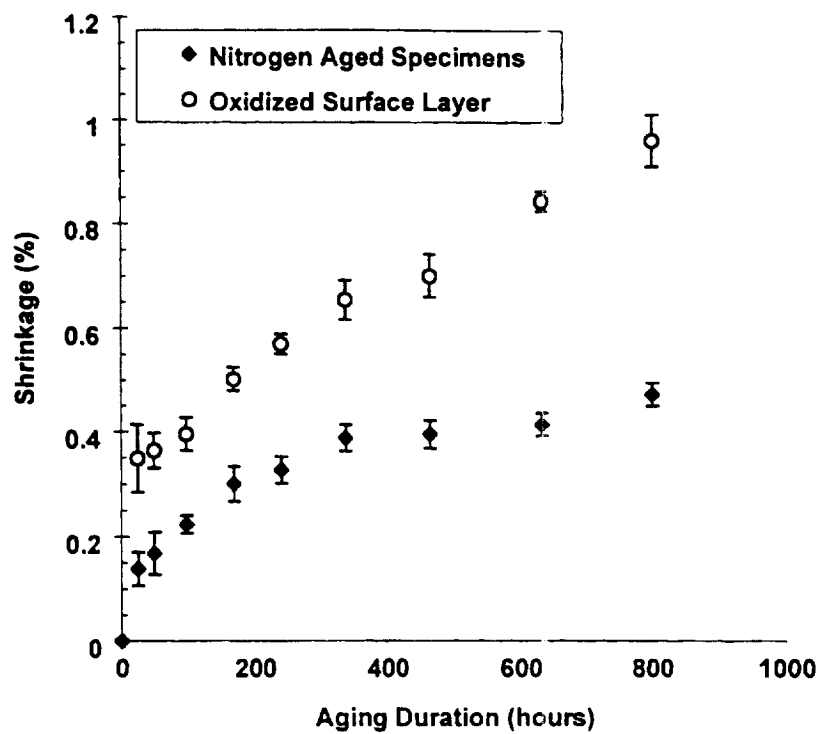Figure 7.—Oxidized surface layer thickness versus aging duration.

Figure 8.—Bend modulus versus aging duration.

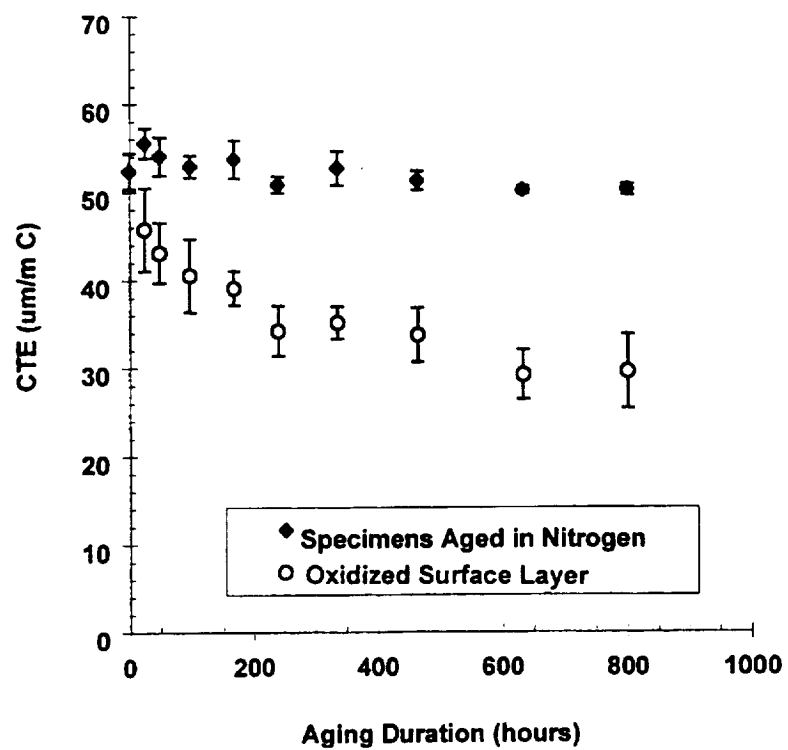Figure 9.—Shrinkage versus aging duration. (2nd paragraph page 7)

Figure 10.—CTE versus aging duration. (3rd paragraph page 7)

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13. ABSTRACT *(Maximum 200 words)*

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